

## SYNTHETIC FLUOR-MAGNESIO-RICHTERITE

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## ABSTRACT

Chemical, physical, optical and x-ray data are provided for fluor-magnesio-richterite, a fluoramphibole synthesized by melt and by solid state reactions. Chemical analysis reveals a unit cell content of  $2(\text{Na}_{1.97}\text{Mg}_{6.01}\cdot\text{Si}_{7.97}\text{O}_{22.04}\text{F}_{1.96})$ . Physical and optical properties are: perfect cleavage  $(110)\wedge(1\bar{1}0)$   $55^{\circ}33'$ ; density  $2.994 \text{ gm. cm.}^{-3}$ ; biaxial(-),  $\alpha=1.5756$ ,  $\beta=1.5894$   $\gamma=1.5954$ ,  $c\wedge Z$   $11.4^{\circ}$ . Unit cell dimensions are  $a$   $9.929 \text{ \AA.}$ ,  $b$   $17.914 \text{ \AA.}$ ,  $c$   $5.274 \text{ \AA.}$ ,  $\beta$   $108^{\circ}16'$ , space group  $I2/m$ .

## INTRODUCTION

Synthetic asbestos investigations at the U. S. Bureau of Mines, Norris, Tennessee, have resulted in the synthesis of more than 75 fluoramphiboles of varied composition, many being fluoride analogs of the natural hydroxyl amphiboles—tremolite, edenite, richterite, and eckermanite. The syntheses and physical properties of these fluoramphiboles have been reported by Eitel (1952), Comeforo and Kohn (1958), Kohn and Comeforo (1955), and Shell, Comeforo, and Eitel (1958). Described here is a fluoramphibole referred to as fluor-magnesio-richterite ( $\text{Na}_2\text{Mg}_6\text{Si}_8\text{O}_{22}\text{F}_2$ ) which differs from fluor-richterite ( $\text{Na}_2\text{CaMg}_5\text{Si}_8\text{O}_{22}\text{F}_2$ ) by the substitution of an additional  $\text{Mg}^{2+}$  for  $\text{Ca}^{2+}$ . When synthesized by pneumatolytic action is formed as an aggregate of acicular crystals, some of which approached fiber dimensions.

## CHEMICAL SYNTHESIS

Fluor-magnesio-richterite was synthesized both from a melt and by pneumatolysis. The theoretical formula is  $\text{Na}_2\text{Mg}_6\text{Si}_8\text{O}_{22}\text{F}_2$ , however, batch compositions containing more fluoride were actually used. The excess fluoride provided for loss of volatiles and promoted reactivity. The batch composition  $\text{Na}_2\text{Mg}_6\text{Si}_8\text{O}_{21.5}\text{F}_3$ , when melted and crystallized, yielded acicular fluoramphibole as the major phase. The batch compositions (1)  $\text{Na}_2\text{Mg}_6\text{Si}_8\text{O}_{21}\text{F}_4$ , (2)  $\text{Na}_2\text{Mg}_6\text{Si}_8\text{O}_{20.5}\text{F}_5$ , and (3)  $\text{Na}_2\text{Mg}_6\text{Si}_8\text{O}_{19.5}\text{F}_7$  were reacted by pneumatolysis. Typically the pneumatolytic product consisted of a mass of short interpenetrating needles considerably less in diameter than those obtained from the melt.

Fluor-magnesio-richterite is the most acicular of any fluoramphibole synthesized by us at this laboratory. In fact, some of the crystals synthesized by pneumatolytic and solid state reaction approached fiber dimensions; they were commonly too small in diameter to be resolved optically at  $450\times$ .

One non-stoichiometric batch composition (for amphibole) gave excellent single crystals for the  $x$ -ray and optical study. It was  $\text{Na}_{6.4}\text{Mg}_{8.6}\text{Si}_8\text{O}_{20.14}\text{F}_{12.8}$ . From this melt fluor-magnesio-richterite and  $\text{NaMgF}_3$  were the major products. This composition was reacted because of a report by Saito (1955) that an orthorhombic amphibole was obtained therefrom. We, however, found only the monoclinic amphibole described herein.

Batch materials were: analytical reagent grade  $\text{NaF}$  and  $\text{Na}_2\text{CO}_3$ , technical grade  $\text{MgO}$  and  $\text{MgF}_2$ , and  $-200$  mesh quartz that was 99.8 per cent  $\text{SiO}_2$ .

The properly proportioned batch materials were thoroughly admixed. For synthesis by melting, the blended raw materials were fired in a sealed graphite crucible which was placed inside a fire-clay crucible to prevent oxidation. After 2 hours at  $1250^\circ\text{C}$ ., the temperature was lowered to below the solidus at  $10^\circ\text{C}/\text{hour}$ . Either graphite or platinum crucibles prevented contamination of the melt; both types were used. For synthesis by pneumatolytic-solid state reaction, fire-clay crucibles alone were satisfactory.

#### CHEMISTRY

The chemical analysis (Table 1, col. 2) was obtained on crystals selected from three separate reaction products. The crystals were selected under the binocular microscope and purified by electro dialysis at 400 volts for 19 hours.

The empirical unit cell content of fluor-magnesio-richterite was found by determining the mass in a mole of unit cells and allocating this mass to the various oxides on the basis of their respective percentages reported in the chemical analysis. The density of the fluoramphibole,  $\rho = 2.994\text{ gm. cm.}^{-3}$  at  $27^\circ\text{C}$ ., was measured on single crystals using the Roller-Smith precision balance with toluene as the suspension medium. The unit cell volume  $V$  was calculated from the  $x$ -ray powder diffraction data to be  $890.8067\text{ \AA}^3$ . Substituting these values in the relation  $ZM = \rho V/1.66020$ , the mass in a mole of unit cells ( $ZM$ ), was determined to be 1606.47 (Table 1). Assuming  $Z$  (the number of formula units for clinoamphibole) to be 2, the chemical formula is determined, except for the oxygen, by dividing the value in column 3 by the values in column 4 (except for  $\text{Na}_2\text{O}$ , the number for which was doubled to yield Na). The oxygen was, however, readily calculated according to the principle of electrostatic neutrality and the formula for fluor-magnesio-richterite becomes  $\text{Na}_{1.97}\text{Mg}_{6.01}\text{Si}_{7.97}\text{O}_{22.04}\text{F}_{1.96}$ . A normalization of the chemical analysis on the basis of 24 ions after Shell and Craig (1956) results in the formula  $\text{Na}_{1.98}\text{Mg}_{6.03}\text{Si}_{7.99}\text{O}_{22.04}\text{F}_{1.96}$ . The two unit formulae, each calculated on a different basis, show good agreement.

TABLE 1. CALCULATION OF THE UNIT CELL CONTENT  
Analyst, R. E. Hooper

	Analysis wt. %	Mass units per mole of unit cells	Molecular wt.	Number of ions in the unit cell
SiO <sub>2</sub>	59.30	957.33	60.09	15.95
MgO <sup>2</sup>	30.00	484.31	40.32	12.02
Na <sub>2</sub> O	7.55	121.89	61.98	3.94
F	4.60	74.26	19.00	3.908
	101.45	1637.79		
Less O=F	-1.94	-31.32		
Total	99.51	1606.47		

## GONIOMETRIC DATA

Crystals studied with the Eichner two circle goniometer are typically acicular in the *c*-axis direction but were rarely terminated. The angle (110)∧(110) is 55°33' (average of four measurements from different crystals). Calculation of this angle from the *x*-ray data yields 55°30'. For the angle (110)∧(100) the measured angle is 62°12' and the calculated angle 62°15'. Perfect cleavage is parallel to the *c*-axis.

## OPTICAL CONSTANTS

The refractive indices of fluor-magnesio-richterite were measured using a 5-axis universal stage and the single variation technique and were plotted on Hartmann dispersion nets. Straight lines were fitted to the plots by a non-statistical method for the purpose of averaging the measured values. The refractive indices given in Table 2 were obtained directly from the Hartmann nets by reading the intersection of the dispersion lines of the grains with the F, D, and C Fraunhofer lines. The accuracy of these results is believed to be ±0.0005.

TABLE 2. OPTICAL CONSTANTS FOR FLUOR-MAGNESIO-RICHTERITE

A	$\gamma$	$\beta$	$\alpha$	$\gamma-\alpha$	$\gamma-\beta$	Optic Sign	2V* (calc.)
F(4861)	1.6007	1.6952	1.5850	.0157	.0055	(-)	72.1
D(5893)	1.5954	1.5894	1.5756	.0198	.0060	(-)	67.8
C(6563)	1.5933	1.5869	1.5718	.0215	.0064	(-)	65.8
N <sub>F</sub> -N <sub>c</sub>	.0074	.0083	.0132				

\* By direct measurement 2V=70.2 using a white light source;  $c/\wedge Z=11.4^\circ$ .

## SINGLE CRYSTAL DATA

A clear, single crystal of fluor-magnesio-richterite was mounted on the precession instrument, and the first two or three reciprocal lattice layers along [100], [010] and [001] were photographed. All spectra recorded were consistent with the diffraction rules of the space group  $I2/m$  as reported for clinoamphibole by Warren (1930).

## POWDER DIFFRACTION DATA

The x-ray powder diffraction data were obtained using a Norelco high-angle counter diffractometer operated in conjunction with an automatic strip chart recorder. Crystals of the fluoramphibole were optically selected, ground under acetone, and the fraction passing the  $-400$  screen was used in making the smear mounts to be x-rayed. The smear mounts were prepared by admixing the powder with a finely ground internal standard and adhesive lacquer, and spreading the resultant slurry as a uniformly thin, translucent film on a BT-cut quartz oscillator plate. The use of the quartz plate is believed to minimize the background radiation thereby permitting a more precise location of the  $2\theta$  maxima (Buerger and Kennedy, 1958). The lacquer consisted of about 10 parts duco-cement dissolved in about 100 parts acetone. An internal standard, a synthetic spinel  $MgAl_2O_4$  calibrated by fixed counting diffractometer techniques to transistor grade silicon  $a = 5.4306 \text{ \AA}$ , was used to correct the diffraction peaks of the fluor-magnesio-richterite and to eliminate, in general, the systematic errors inherent in the diffractometer method.

The settings of the diffractometer were: divergence and scatter slits,  $1^\circ$ ; receiving slit, 0.006 inches; scanning speed,  $\frac{1}{4}$  degree  $2\theta$  per minute; strip chart scale,  $\frac{1}{2}$  degree per inch; time constant, 4; multiplier, 1; scale factor, 16; and Ni filtered Cu radiation,  $K_\alpha = 1.54178 \text{ \AA}$ ,  $K_{\alpha_1} = 1.54051 \text{ \AA}$ .

The  $2\theta$  values recorded on the diffraction charts were located by measuring the peak centers at two-thirds height as proposed by Donnay and Donnay (1951). This procedure of peak location was used, however, only for peaks showing completely resolved  $\alpha_1$  and  $\alpha_2$  spectral components. For unresolved or partially resolved peaks, the peaks were located by measuring their centers at half height (Smith and Sahama, 1954). The diffraction peaks were then corrected to the internal standard, and comparable  $2\theta$  values from four separate strip charts were averaged and converted to quadratic values from the tables of Gibbs and Lewis (1961). The assignment of diffraction indices to the powder diffraction record was made by comparison with the Kohn and Comeforo (1955) data for synthetic fluor-richterite and by use of the nomogram of Bloss and Gibbs (1961). The final indexing of the record was confirmed by visually comparing the relative intensities of the indexed spectral components re-

corded on the precession films with those of comparable powder diffraction peaks. The dimensions of the unit cell were found by a least squares refinement of forty-four reflections of the powder pattern as  $a=9.929 \text{ \AA}$ ,  $b=17.914 \text{ \AA}$ ,  $c=5.274 \text{ \AA}$ ,  $\beta=108^\circ 16'$ , all  $\pm 0.05$  per cent, space group

TABLE 3. POWDER DIFFRACTION DATA

hkl	I/I <sub>0</sub>	Observed Values <sup>1</sup>			Calculated Values <sup>1</sup>			$\Delta Q \times 10^5$
		d	$2\theta$	$Q \times 10^5$	d	$2\theta$	$Q \times 10^5$ <sup>2</sup>	
020	4	8.960	9.870	1,245	8.956	9.875	1,246	-1
110	58	8.346	10.597	1,435	8.343	10.600	1,436	-1
011	14	4.825	18.385	4,295	4.824	18.390	4,297	-2
200	21	4.720	18.798	4,488	4.716	18.818	4,497	-9
040	24	4.481	19.809	4,979	4.478	19.824	4,986	-7
21 $\bar{1}$	16	4.033	22.036	6,147	4.034	22.035	6,146	+1
031	39	3.840	23.170	6,786	3.837	23.177	6,790	-4
23 $\bar{1}$	52	3.404	26.191	8,639	3.403	26.193	8,640	-1
150	11	3.353	26.587	8,897	3.351	26.614	8,915	-18
240	75	3.249	27.453	9,475	3.247	27.466	9,483	-8
310	100	3.099	28.813	10,416	3.097	28.834	10,431	-15
32 $\bar{1}$	40	2.964	30.150	11,383	2.964	30.145	11,379	+4
051	26	2.916	30.663	11,764	2.915	30.679	11,776	-12
330	27	2.784	32.150	12,902	2.782	32.179	12,924	-22
25 $\bar{1}$	92	2.711	33.048	13,612	2.709	33.065	13,626	-14
231	18	2.680	33.427	13,918	2.678	33.450	13,936	-18
16 $\bar{1}$	30	2.581	34.726 $\alpha$ 1	15,011	2.582	34.718	15,004	+7
002	27	2.505	35.856	15,944	2.505	35.864	15,940	+4
170	<5	2.469	36.380	16,398	2.469	36.374	16,393	+5
350	21	2.361	38.078 $\alpha$ 1	17,936	2.363	38.049	17,910	+26
251	27	2.300	39.157	18,895	2.299	39.185	18,922	-27
071	22	2.279	39.533	19,246	2.279	39.542	19,254	-8
112	7	2.233	40.359 $\alpha$ 1	20,058	2.235	40.323	20,024	+34
332	<5	2.188	41.264	20,893	2.186	41.257	20,921	-28
27 $\bar{1}$	5	2.178	41.444	21,068	2.177	41.445	21,104	-36
36 $\bar{1}$	30	2.163	41.716 $\alpha$ 1	21,368	2.164	41.699	21,351	+17
132	<5	2.108	42.869 $\alpha$ 1	22,509	2.107	42.877	22,517	-8
202	<5	1.972	45.989 $\alpha$ 1	25,722	1.971	46.001	25,734	-12
190	<5	1.948	46.583 $\alpha$ 1	26,353	1.947	46.595	26,365	-12
062	7	1.916	47.397 $\alpha$ 1	27,229	1.919	47.332	27,158	+71
510	13	1.876	48.470 $\alpha$ 1	28,400	1.875	48.488	28,421	-21
091	8	1.849	49.234 $\alpha$ 1	29,246	1.850	49.215	29,226	+20
530	7	1.797	50.755 $\alpha$ 1	30,960	1.799	50.714	30,914	+46
291	<5	1.659	55.342 $\alpha$ 1	36,350	1.658	55.360	36,372	-22
480	13	1.623	56.647 $\alpha$ 1	37,941	1.624	56.640	37,933	+8
1.11.0	6	1.605	57.339 $\alpha$ 1	38,795	1.605	57.367	38,830	-35
253	9	1.577	58.494 $\alpha$ 1	40,235	1.577	58.475	40,211	+24
600	20	1.571	58.710 $\alpha$ 1	40,506	1.572	58.687	40,477	+29
65 $\bar{1}$	7	1.503	61.667 $\alpha$ 1	44,279	1.502	61.691	44,310	-31
402	<5	1.499	61.859 $\alpha$ 1	44,528	1.499	61.858	44,527	+1
0.12.0	17	1.493	62.120 $\alpha$ 1	44,867	1.493	62.125	44,873	-6
3.11.0	<5	1.446	64.394 $\alpha$ 1	47,853	1.446	64.372	47,825	+28
56 $\bar{1}$	16	1.415	65.942 $\alpha$ 1	49,921	1.415	65.951	49,932	-11
563	7	1.344	69.922 $\alpha$ 1	55,345	1.344	69.916	55,336	+9
0.12.2	5	1.282	73.877 $\alpha$ 1	60,873	1.282	73.835	60,813	+60

<sup>1</sup> Values for CuK $\alpha$  unless otherwise indicated; CuK $\alpha$  1.54178  $\text{\AA}$ , CuK $\alpha_1$ , 1.54050  $\text{\AA}$ .

<sup>2</sup> Based on the following lattice constants:  $a=9.929 \text{ \AA}$ ,  $b=17.514 \text{ \AA}$ ,  $c=5.274 \text{ \AA}$ ,  $\beta=108^\circ 14'$ , space group  $I2/m$ .

I2/m. Upon transformation to the C2/m orientation using the matrix  $101/010/00\bar{1}$  set forth by Zussman (1959), the unit cell dimensions become  $a=9.677 \text{ \AA}$ ,  $b=17.914 \text{ \AA}$ ,  $c=5.274 \text{ \AA}$ ,  $\beta=102^\circ 57'$ , all  $\pm 0.05$  per cent.

#### DISCUSSION

The general formula for amphibole may be given as  $W \cdot X_2 \cdot Y_5 \cdot Z_8 \cdot O_{22} \cdot (OH, F)_2$  where W refers to ions of radius 0.7 to 1.3  $\text{\AA}$  which can occupy the A site at  $(\frac{1}{2}, 0, \frac{1}{2})$  in the structure (Whittaker, 1960), chiefly  $K^+$ ,  $Na^+$ ,  $Ca^{2+}$ ,  $Mg^{2+}$  and  $Li^+$ ; X refers to ions of radius 0.7 to 1.1  $\text{\AA}$  which occupy the  $M_4$  sites at the edges of the amphibole chains, chiefly  $Ca^{2+}$ ,  $Na^+$ ,  $Fe^{2+}$ ,  $Mg^{2+}$ ,  $Li^+$ , and  $Mn^{2+}$ ; Y refers to ions of radius 0.5 to 0.9  $\text{\AA}$  sandwiched in six-fold coordination between the chains, chiefly  $Mg^{2+}$ ,  $Fe^{2+}$ ,  $Mn^{2+}$ ,  $Fe^{3+}$ ,  $Al^{3+}$ ,  $Li^+$ , and  $Ti^{4+}$ ; and Z refers to small, high valence ions of four-fold coordination in the chains, principally  $Si^{4+}$  but to a lesser extent (up to 25 per cent)  $Al^{3+}$ .

Crystal chemical principles permit a likely assignment of sodium and magnesium to sites in flour-magnesio-richterite. As Whittaker (1960) has shown, the average radius of the ions filling the  $M_4$  sites in clin amphibole controls in general the value of the crystallographic angle  $\beta$ ; that is, larger ions reduce the displacement between the neighboring  $(Si_4O_{11})$  chains and thereby the magnitude of  $\beta$ . Whittaker demonstrated this relation in a regression plot of the  $\beta$  angles on the average radii of the ions filling the  $M_4$  sites for five structurally determined amphiboles and for amosite (Fig. 1). Since Whittaker published this regression, structural

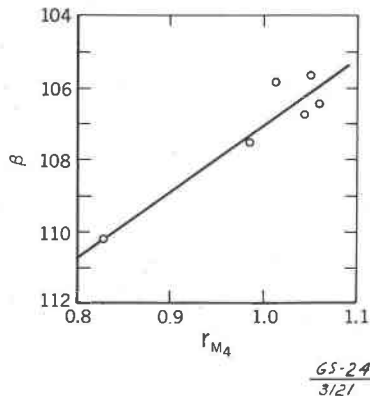


FIG. 1. Plot of  $\beta$  against the mean Goldschmidt radius of the ion at  $M_4$  for the five clin amphiboles whose structures have been published, and for amosite. The line is the regression line of  $\beta$  on  $r$  (After Whittaker).

analyses for grunerite and cummingtonite have been carried out by Ghose (1959, 1961). Employing the average radii of the ions filling the  $M_4$  sites in these amphiboles, we read the  $\beta$  angles from Whittaker's regression lines as  $109^\circ 59'$  and  $110^\circ 12'$  which agree remarkably well with Ghose's experimental values of  $\beta$ ,  $109^\circ 50'$ , and  $109^\circ 54'$ , respectively. To be consistent with the regression plot, it was necessary to transform the cell setting of the cummingtonite from a C-face centered cell as given by Ghose to a I-centered cell.

In fluor-magnesio-richterite, sodium and magnesium ions differ sufficiently in radii that a filling of the  $M_4$  sites by one rather than the other of these ions should cause an appreciable difference in  $\beta$ . Since  $\text{Na}^+$  tends to fill only the A and  $M_4$  sites in amphibole (Whittaker, 1949; Heritch, *et al.*, 1957, 1960a, 1960b) one can give, in general, three possible distributions of  $\text{Mg}^{2+}$  and  $\text{Na}^+$  in fluor-magnesio-richterite: (1)  $\text{Mg}^{2+}$  filling the A site and  $2\text{Na}^+$  the  $M_4$  sites; (2)  $\text{Na}^+$  filling the A site and both  $\text{Na}^+$  and  $\text{Mg}^{2+}$  the  $M_4$  sites; or (3)  $\text{Mg}^{2+}$  and  $\text{Na}^+$  randomly filling the A and  $M_4$  sites. Depending upon whether (1), (2), or (3) pertains, the average radius for the ions filling the  $M_4$  sites would be (1) 0.98, (2) 0.88 and (3) 0.91 Å, respectively. The  $\beta$  angles read from Whittaker's regression line for each of these possibilities are  $107^\circ 24'$ ,  $109^\circ 08'$  and  $108^\circ 36'$ . The measured  $\beta$  angle for fluor-magnesio-richterite is  $108^\circ 16'$  which suggests that the third possibility is more correct, namely,  $\text{Na}^+$  and  $\text{Mg}^{2+}$  are randomly distributed in the A and  $M_4$  sites. If this is true, then the formula for fluor-magnesio-richterite should be given as  $\text{Na}_2\text{MgMg}_5\text{Si}_8\text{O}_{22}\text{F}_2$ . However, this particular distribution of ions would be very difficult to verify by an x-ray structural analysis because of the similarity in electron density of  $\text{Na}^+$  and  $\text{Mg}^{2+}$ .

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